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COMPLETE SPECIFICATION.

Process for Producing, Decomposing, Converting, or Transforming Compounds, and Reducing Metallic Compounds so Generated to Metallic State.

I, HENRY SPENCER BLACKMORE, resident of the City of Mount Vernon, County of West Chester, State of New York, United States of America, and by occupation a Chemist, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

My invention relates to a process for producing, decomposing, converting, or transforming metallic compounds; and reducing same to metallic state together with the production of materials employed to produce such transformation and reduction, and the mode of their application, representing various steps in a complete process for reduction of metals from natural products or compounds particularly relating to those of an infusible or semi-fusible character, such as those of the earth and alkali earth metals.

The object of my invention is to produce in an economical manner, purified chemical compounds, from natural products chiefly of an infusible or semi-fusible nature, and finally reducing the same to metallic state when so desired; and has particular reference to the production of sulphides of such metals as aluminium, magnesium, calcium, strontium, barium *etc.*, and their reduction finally to metallic state by action of molten or highly heated reducing agents as hereinafter described but it is not limited to them as many other compounds or metals may be likewise produced, and consists essentially in dissolving or, in case of producing sulphides, suspending, the infusible or semi-fusible oxides, or other compounds of such metals, in a molten bath and exposing them in a heated condition to the action of carbon disulphide vapor in which condition the oxide is transformed into sulphide with the elimination of carbonic anhydride and carbon oxy-sulphide, then finally if so desired converting the sulphides into metallic form by treatment with highly heated or molten material or elements having greater affinity for sulphur than the metal of sulphide thus produced, and preferably of greater specific gravity than the molten bath of sulphides.

Among the oxides thus convertible to sulphides are alumina, magnesia, lime, strontia and baryta, and reducing agents capable of being employed under various conditions of affinity are lead, copper, zinc, iron, potassium cyanide *etc.*, in highly heated or molten condition preferably in state of fine sub-division.

As an illustration of the manner in which I carry out this process on a practical basis, I will take for example the production of aluminium sulphide from natural bauxite and reduction of the same to metallic aluminium.

The natural product, bauxite, employed consists of oxides of aluminium and iron contaminated more or less with silica, this bauxite is first thoroughly dried and pulverized, it is then dissolved in the molten bath consisting of fluorides and sulphides of potassium and sodium in about equal portions, the object of using potassium and sodium salts mixed is to produce a material melting at as low temperature as possible, the mixture of alkali salts melting at a lower temperature than either of them separate and independent; it is obvious that the molten bath may consist of various other substances which are capable of dissolving the infusible or semi-fusible oxide treated, or sulphide produced, but have found the mixture of alkali fluorides and sulphides to be the most efficient.

When the molten bath has become saturated with bauxite, apparatus is closed and

[Price 8d.]

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a mixture of carbon disulphide and sulphur fluo-carbide vapor is passed through the same. The fluorine of the fluo-carbide combines with silicon (contaminating the bath) converting the silica into gaseous silicon tetra-fluoride, and remaining carbon and sulphur combine with oxygen of silica producing carbon oxy-sulphide, or carboxyl sulphide, which together with silicon tetra-fluoride escapes from the apparatus, leaving the dissolved or suspended oxides free from silicon. The carbon di-sulphide at the same time converts the alumina or aluminium oxide into sulphide producing simultaneously carbonic anhydride and carbon oxy-sulphide or carboxyl sulphide which escape in gaseous form. The same action takes place converting the iron oxide present into sulphide.

I am fully aware that dry infusible aluminium oxide (alumina) has been converted into aluminium sulphide by passing vapor of carbon di-sulphide over it in its heated state but in that case the particles of aluminium oxide being infusible at the temperature employed, is only transformed superficially into sulphide which being fusible coats said particles thus preventing further action, the richest product obtainable by this means being about 35 *per cent.* sulphide, the balance being unconverted alumina, a process too expensive to be operated in a commercial way, on account of great waste of aluminium oxide and difficulty of reducing said sulphide on account of infusibility of the mass, being almost impossible to reach sulphide with reducing agents, and when reduced to metal; leaving same disseminating throughout the material in such a manner that it is impossible to combine reduced metals in a button; by dissolving oxide in molten bath there are no particles to coat, or by suspending the same in molten bath capable of dissolving the sulphide produced, thus exposing fresh surfaces of oxide to action according to this step of the process described, the difficulty is thus entirely obviated.

The mixture of sulphides dissolved or suspended in the molten bath, produced as aforesaid is exposed to the action of molten or highly heated iron or copper in a finely divided state, which having greater affinity in its heated state, for the sulphur combined with the aluminium, absorbs the same producing iron or copper sulphide and free metallic aluminium which being lighter than the surrounding bodies ascends and accumulates on the top of the bath from whence it is withdrawn at the end of the process and cast into ingots.

The iron or copper sulphide thus produced being less soluble in molten bath remains suspended therein so long as reduction goes on, after which it gradually settles and becomes molten at the bottom of the retort from which it is first drawn, this being followed by the molten flux or bath of alkali fluoride and sulphides which is afterward used over to dissolve more bauxite as before, and finally the aluminium is withdrawn and cast into ingots.

The iron may be also introduced into the molten sulphide in a finely divided state such as is known in commerce as "iron by hydrogen" this fine iron being first heated to a bright red heat and gradually sifted or conveyed into the molten bath the same reaction taking place as with the molten iron.

When molten iron is employed it is first passed through a firebrick sieve so that the metal will be introduced into sulphide bath in fine streams or of fine shot formation to admit of complete sulphurization of iron before reaching bottom of retort, which is therefore preferably of considerable depth.

The carbon di-sulphide and sulphur fluo-carbide employed to transform oxides into sulphides and remove silica is produced by heating carbon (charcoal) together with a fluoride (such as lead fluoride) in a retort, to a low red heat, and then passing the vapor of sulphur through the same, when the products distill, and are passed through the bath as before stated.

It is obvious that any other liquid or volatile combination of carbon with other electro-negative elements may be employed to convert the metallic oxide into other binary compounds applicable for economical reduction, such as phosphorus tellurium, selenium, *etc.*, without departing from the spirit of my invention, which consists essentially in converting metallic oxides or other infusible or semi-fusible

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compounds into compounds with other elements of electro-negative nature, by dissolving or suspending the same in a molten bath and exposing it to action of compounds of carbon and the other element a compound of which is desired, such as carbon sulphide when sulphide is desired, and finally reducing said compound produced in the bath by such action to metallic state, by mixing with a heated element or compound having greater affinity for the electro-negative element combined in the compound so produced, substantially as described.

Aluminium sulphide can also be reduced to metallic state by treatment with potassium cyanide, the potassium cyanide absorbs sulphur producing potassium sulpho-cyanide or thio-cyanate, care must be taken in this case not to have the temperature too high otherwise the sulpho-cyanide will be dissociated into potassium mellonide; potassium sulphide, free sulphur and carbon, in which case more or less of the aluminium will be destroyed by free sulphur and bath saturated with fine carbon so that it is almost impossible to combine metal into a mass or

button.

By dissolving a mixture or compound of silicon and aluminium oxides, preferably certain clays (aluminium silicate) in molten bath aforesaid, and then exposed to the action of carbon di-sulphide vapor alone, ultramarine may be produced which can be finely separated from soluble constituents of molten bath by lixiviation and filtration or decantation; the material preferably employed for this purpose is kaolin, a clay consisting of a mixture of aluminium silicate and undecomposed feldspar. The retorts employed are lined with carbon to protect the same from solvent action of bath.

Alkali cyanides may also be produced by suspending iron carbide in molten alkali sulphides and passing nitrogen gas through the molten bath producing at the same time iron sulphide.

I do not claim the dissolving or suspending of various infusible or semi-fusible substances treated under this process but simply the process of acting upon or transforming them while thus dissolved or suspended by means of elements or compounds brought in contact with the same, from a foreign source, and principally relates to those substances which do not combine directly with molten bath or solvent, forming definite compounds, except during introduction of the active agent from a foreign source such as carbon di-sulphide in formation of aluminium sulphide *etc.*, as hereinbefore described.

Also alkali carbonates or bi-carbonates may be produced from natural alkali chlorides; such as sodium carbonate from common salt (sodium chloride). By suspending alumina (aluminium oxide) in molten bath of salt in closed receptacle and passing superheated steam through the molten mass whereby the material is transformed into sodium aluminate and hydrochloric acid which is conducted from the apparatus when this re-action is complete, the steam is shut off and carbon di-oxide is introduced in its stead when the sodium aluminate is transformed into the sodium carbonate or bi-carbonate and free alumina which is used over.

I am aware that sodium silicate has been produced by mixing silica with common salt (sodium chloride) and melting the same and then exposing to the action of steam thereby eliminating hydrochloric acid but in that case the silica combines directly with the molten compound when heated, forming sodium chlor-silicate which by action of steam is decomposed into sodium silicate and hydrochloric acid in which case it is readily seen that the silica is not dissolved or suspended in the molten salt, but combined with the same, forming a definite compound, so that when the steam is introduced from a foreign source, it reacts on compound thus produced and not the silica *per se* dissolved or suspended in molten mass, hence the difference from my process can be readily seen.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. The process for producing a fluo-carbide which consists in exposing carbon and

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metallic fluoride in a heated state to the action of a volatile element (such as sulphur, tellurium, selenium, *etc.*) a compound of which is desired.

2. The process for producing compounds of carbon and fluorine, with other elements, which consists in passing the vapor of such elements over a mixture of carbon and metallic fluoride, (such as lead fluoride) in a heated state, substantially as described. 5

3. The process for producing sulphur fluo-carbide which consists in passing the vapor of sulphur over a heated mixture of carbon and metallic fluoride, substantially as described. 11

4. The process for eliminating silicon from a molten bath, containing the same which consists in exposing said molten bath to the action of fluorine carbide or compound of same, substantially as described. 10

5. The process for eliminating silicon from a molten bath or compound containing the same, which consists in exposing said bath to the action of sulphur fluo-carbide, substantially as described. 16

6. The process for eliminating silicon from a molten bath containing the same which consists in exposing said bath to the action of a non-metallic fluo-carbide in the form of vapor, substantially as described.

7. The process for converting metallic oxides into other compounds which consists in the dissolving or suspending said oxides in a molten bath and exposing the same to the combined action of carbon and sulphur or other element, a compound of which is desired, substantially as described. 20

8. The process for producing, decomposing, converting, or transforming compounds of an infusible or semi-fusible nature which consists in dissolving or suspending the same in a molten bath and exposing it in a heated state to the action of electro-negative elements or compounds of the same, substantially as described. 25

9. The process for producing, decomposing, converting, or transforming metallic oxides which consists in bringing said oxides to molten or liquified state and exposing it to the action of electro-negative elements or compounds of the same, substantially as described. 30

10. The process for producing aluminium or other sulphide which consists in exposing the molten or liquified oxides to the action of carbon di-sulphide vapor, substantially as described.

11. The process for producing aluminium sulphide, which consists in dissolving or suspending aluminium oxide in a molten bath and exposing the same in its heated state to the combined action of carbon and sulphur, substantially as described. 35

12. The process for producing aluminium sulphide, which consists in dissolving or suspending aluminium oxide in a molten bath and passing the vapor of carbon di-sulphide over or through the same, substantially as described.

13. The process for producing a cyanide, which consists in suspending a metallic carbide in a molten bath and exposing the same to the action of nitrogen gas, substantially as described. 40

14. The process for producing alkali cyanide which consists in suspending carbide of iron in a molten bath of alkali sulphide and introducing nitrogen gas into the mixture, substantially as described.

15. The process for producing alkali salts from natural products, such as sodium chloride, which consists in suspending or dissolving alumina in a molten bath of said natural product and exposing the same to the alternate action of steam and gaseous acid, a compound of which is desired, substantially as described. 45

16. The process for producing alkali carbonate, which consists in dissolving or suspending alumina in molten bath of alkali chloride and subjecting the same to the action of super-heated steam, thereby eliminating chlorhydric acid and producing alkali aluminate, and finally introducing carbonic anhydride, substantially as described. 50

17. The process for producing ultramarine which consists in dissolving kaolin or feldspar in molten bath and exposing same to action of carbon di-sulphide vapor, substantially as described. 55

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18. The process for reducing metals from their salts or combinations which consists in dissolving the same in a molten bath and gradually exposing it to the action of highly heated or molten reducing agents, substantially as described.
19. The process for producing aluminium from its compound which consists in
5 dissolving said compound in a molten bath and gradually introducing in small streams metallic iron or copper in a highly heated or molten state, substantially as described.
20. The process for reducing aluminium from its sulphides which consist in
10 exposing the molten or liquified sulphide to the action of finely divided iron or copper in heated state, substantially as described.
21. The process for reducing aluminium, or other metals which consists in gradually introducing reducing agents, in a finely divided heated state into a molten bath containing dissolved compounds of the metal desired, substantially as described.
- 15 22. The process for reducing aluminium or other metals from their combinations which consists in dissolving said combinations in a molten bath, and gradually introducing highly heated reducing agents in a fine state of sub-division into the same, substantially as described.
23. The process for reducing aluminium or other metals from their compounds.
20 which consists in bringing said compounds in a molten or dissolved state gradually in contact with a highly heated molten reducing agent, substantially as described.
24. The process for reducing aluminium or other metals which consists in slowly commingling said compounds in a molten or dissolved state with a highly heated or molten reducing agent of greater specific gravity, substantially as described.
- 25 25. The continuous process for producing aluminium or other metals which consists in exposing the molten or suspended oxides to the alternate action of a non-metallic carbide and a highly heated or molten reducing agent of greater specific gravity, substantially as described.
26. The continuous process for producing aluminium and other metals which
30 consists in exposing molten metallic oxides to the alternate action of a non-metallic carbide and a molten compound, (such as potassium cyanide) capable of absorbing or combining with the combined electro-negative element liberating metal desired, substantially as described.
27. The continuous process for producing aluminium or other metal which
35 consists in dissolving or suspending oxides of such metals in molten bath and exposing the same to the alternate action of a non-metallic carbide and highly heated or molten material capable of absorbing or combining with the electro-negative element of the compound so produced, and then withdrawing the products of the reaction and the metal reduced, substantially as described.
- 40 28. The continuous process for reducing aluminium or other metals which consists in exposing the oxide dissolved or suspended in a molten bath to the alternate action of a non-metallic carbide and highly heated or molten reducing agent, substantially as described.
29. In a process for producing compounds and reducing same to metallic state a.
45 molten flux or bath consisting of a mixture of fluorides and sulphides of the alkali metals, potassium and sodium, substantially as described.
30. The process for producing aluminium from bauxite which consists in dissolving the same in a molten bath consisting of fluorides and sulphides of potassium and sodium, and eliminating the silicon from said bath by treatment with sulphur fluo-
50 carbide and then converting the dissolved aluminium oxide into sulphide by the action of carbon di sulphide vapor and finally reducing said molten aluminium sulphide to metallic state by slowly introducing highly heated or molten iron in fine streams at the same time producing iron sulphide, substantially as described.
31. The process for producing aluminium from bauxite which consists in dissolving
55 the same in a molten bath consisting of fluoride and sulphide, of alkali metals, eliminating the silicon therefrom in the form of gaseous silicon tetra-fluoride by exposing in the molten state the action of the compound formed by action of

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sulphur vapor on heated mixture of carbon and heated metallic fluorides converting the remaining de-siliconized molten aluminium oxide into sulphide by action of carbon di-sulphide vapor and finally slowly and gradually exposing the molten sulphide thus produced to the action of a highly heated or molten material having greater affinity for sulphur thereby liberating the metal desired, substantially as described. 5

32. The process for producing alkali aluminate which consists in exposing aluminium oxide (alumina) dissolved or suspended in molten alkali salt, to the action of steam, substantially as described.

33. The process for producing alkali aluminate and hydrochloric acid which consists in exposing aluminium oxide (alumina) dissolved or suspended in molten alkali chloride to the action of steam, substantially as described. 10

34. The process for producing, decomposing, converting or transforming compounds which consists in exposing same while dissolved or suspended in molten bath to the action of reagents or material introduced from a foreign source and particularly of gaseous or vaporous form and being capable of combining or reacting with suspended or dissolved matter or constituents of the bath or both producing new compounds or products, substantially as described. 15

Dated this 10th day of December 1895.

WHEATLEY & MACKENZIE, 20
Agents.

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